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The Effects of Preparation Methods on $TlSr_2Ca_2Cu_3O_{9-\delta}$ Superconductor

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Abstract:

The effects of manufacturing $TlSr_2Ca_2Cu_3O_{9-\delta}$ crystallized materials superconductors were investigated in this paper. The sol-gel and solid-state reaction methods were investigated by evaluating the physical parameters of the superconductor's behaviors. The process duration for sample of Sol-gel requires 12 hours of heat procedure, while the solid-state reaction methods needs 96 hours of duration time. The sintering temperature is 860 °C for both investigated methods. The $TlSr_2Ca_2Cu_3O_{9-\delta}$ superconductor properties such as, Resistance variation with temperature, transformation temperature, X-ray diffraction, Atomic Force Microscope, the values of the oxygen content, lattice parameters. The samples showed very good metal behavior. The, $T_{c\ (R = 0)}$ for Sol-gel samples and solid-state reaction was found to be at 114 K, and 111 K respectively. The results of X-ray diffraction give evidence of orthorhombic structure for all samples, and average diameter 92.36nm and 108.24nm. However, the difficulties and duration process of Sol-gel method is less than solid-state reaction. The lattice parameters (a, and c) showed that sample which preparing by sol-gel technique are larger than prepared by the solid reaction technique, while (c/a) seems to be decreased respectively.

***Keywords**: $ErSr_2Cu_3O_{7-\delta}$, Solid state reaction method, orthorhombic structure, X-ray diffraction data*

Introduction:

The physical properties of superconductors depend on the synthesis methods and the processing techniques used. Progressive research efforts have led to improved sample preparation while providing significant cost and time to produce high quality samples. The development of a complex system such as $TlSr_2Ca_2Cu_3O_{9-\delta}$ superconductor needs a critical approach in order to obtain a high quality product. There are several ways to prepare a ceramic superconductor that includes the traditional solid state path (SSR), the most commonly used and widely used process that is commonly reported in [1-7]. In general, starting materials, for example, oxides of metals, carbonates or other salts are mixed with suitable proportions, homogenous and usually heated at high temperature. However, this technique has many disadvantages such as long treatment times (24-240 hours), high temperature heat treatment, and several cycles of milling operations to achieve higher purity and homogeneity of the samples. In addition, this technique also shows other defects such as individual particle size, volume distribution, slow reaction rate, and tendency to produce contaminants (non-superconducting phase) [8-10].

At present, solution routes such as co-precipitation [11-14], sol-gel method [15], freeze drying, [16] and spray drying have been developed in order to overcome these problems.

In this paper we focus on the preparation of high-temperature $TlSr_2Ca_2Cu_3O_{9-\delta}$ high temperature by sol-gel method and compare its properties with those obtained by solid state technology. In addition, this

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work also discusses how to prepare samples these methods. Systematic investigations of superconductivity properties were performed using DC temperature measurements. We also report X-ray Diffraction XRD and electron microscopy scanning SEM on the same samples

Experimental

Preparation of the $TlSr_2Ca_2Cu_3O_{9-\delta}$compound by Sol-Gel and the recommended state reaction technique. The analytical grade of $Tlco_3$+$SrCo_3$, $Caco_3$and $CuCO_3$was taken as feedstock and was weighted by the equivalent amount and then dissolved in ethanol and Acetic acid diluted. The mixture is placed in a magnetic stirrer at 80 ° C, followed by ignition, combustion occurs within a few seconds, and nano couple powders have been deposed. These powders crush and completely earthy. The component micro powders were then disinfected at 850 ° C for 12 hours for final formation of $TlSr_2Ca_2Cu_3O_{9-\delta}$nanoparticles. The soft powders were then granulated as a joint and compacted in a disk shape (approximately 13 mm in diameter, thickness 1.8 mm - 2.0 mm) using hydraulic pressure under 5 ton / cm^2 pressure.

The samples were performed by a solid-state reaction method. Powders of Tl2O3+SrO, CaO and CuO were grinded with a gete mortar at 40-60 minutes duration time. The quartz tube was used to heat the materials at furnace for calcination. The heating process for the mixture begins from room temperature, at rising rate of 200 °C / hour, to reach 850 ° C with duration time 12 hours, and then cooled back with the same heating rate.

The preparation of samples takes many steps, beginning with the using of mortar to mix the powder for 40 minutes. The 5 ton/cm^2 hydraulic pressure was used to create a samples pellet with 13 mm diameter and about 2 mm thickness. Quartz tube were used in preparing the samples at 860 °C for 96 hours with rising and cooling temperature at rate 200 °C.

DC-probe technique was used to proof the superconductivity behavior of the prepared samples. iodometric calibration method was used to ensure the content of Oxygen [17, 18]. The methods that been used for measuring critical temperatures as well as the parameters a and c were described elsewhere [19-21].

Results and Discussion

The resistance behavior with temperature of $TlSr_2Ca_2Cu_3O_{9-\delta}$HTSC was investigated with the standard four-probe technique. The normal state resistance of all the samples shows metal like behavior with respect to temperature. Figure (1) illustrated the normalized resistivity vs temperature (ρ–T) behavior for both samples that prepared by sol-gel and solid-state reaction methods (SSR). The values of critical transition temperatures at zero resistivity and onset ($T_{c(off)}$ & $T_{c(onset)}$) were calculated for both samples that prepared by the two methods. These temperatures $T_{c(off)}$ were 114, 111 and $T_{c(onset)}$ were 121,118 K when samples were prepared by sol-gel and (SSR), respectively.

All samples under experiment in this research were subjected to total structural characterization by X-ray diffraction technique. Data of X-ray diffraction collected from various samples (samples prepare by sol-gel and solid state reaction methods (SSR) were a tetragonal structure, polycrystalline and correspond to $TlSr_2Ca_2Cu_3O_{9-\delta}$phases. The XRD also shows some impurity phases with vanishingly small concentrations. The representative XRD patterns are shown in figure (1).

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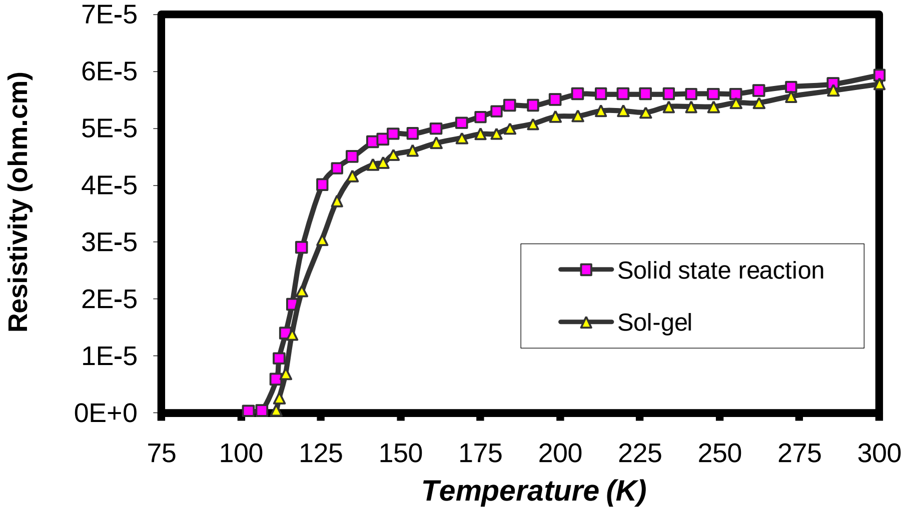

Figure 1. The resistivity vs. temperature for sample the $TlSr_2Ca_2Cu_3O_{9-\delta}$samples prepare by sol-gel and solid-state reaction methods (SSR).

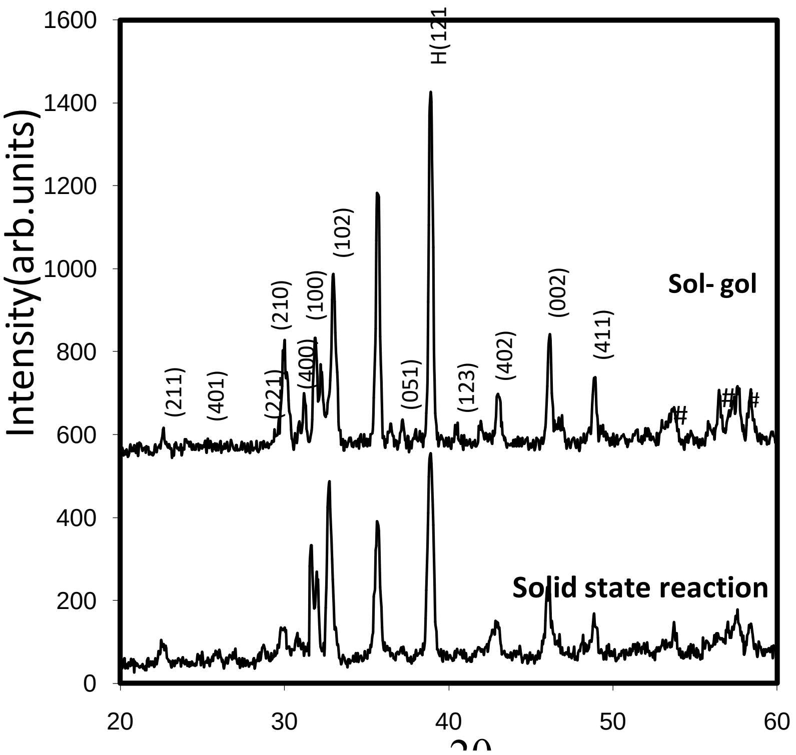

Figure 2. XRD Patterns for the $TlSr_2Ca_2Cu_3O_{9-\delta}$samples for different preparation methods. High phase (1223) reflections and the peaks of $(Ca,Ba)_2CuO_3$ and $CaTlO_4$ are evident with #.

State reaction methods (SSR).

The lattice parameters were estimated using d values and hkl reflections of X-ray diffraction pattern were described elsewhere [19,20]. The values of $T_{c(offset)}$, $Tc_{(onset)}$, oxygen content, excess oxygen $\delta_{(O2)}$, a, c and c/a were calculated, as shown in table-1. The estimated experimental errors are within the rate (± 5%). It was notes from the table that each of the values of the transition temperatures Tc(offset), Tc(onset), oxygen content, parameters of the lattice of the sample which prepared by sol-gel technique is larger than the sample which prepared by the solid

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reaction technique, While the compression of the c/a ratio decreased from 4.522 to 4.519.

Figure 3 shows Atomic Force Microscope AFM image and Granularity accumulation distribution chart of $TlSr_2Ca_2Cu_3O_{9-\delta}$ samples prepare by sol-gel and solid-state reaction methods distributed uniformly on the surface. We noted it was obvious from figure 3and the table that the structure has small ordered particles with semispherical shape. The Avg. Diameter (nm) from sol-gel is 92.36whele it is become 108.24.

In comparaison, the conventional method (solid state method) requiers a multi-binding process and a long heat treatment to obtain a good superconducting phase. Note that the peak density is slightly higher than the samples prepared through Sol-Golhis is probably due to fine powder produced after grinding several during the preparation process. However, the disadvantages that can be seen from SSR technology are, they require a long time Sintering time synchronous with intermittent grinding to improve solid state reaction and to obtain the same quality produced by Sol-Gol technique.

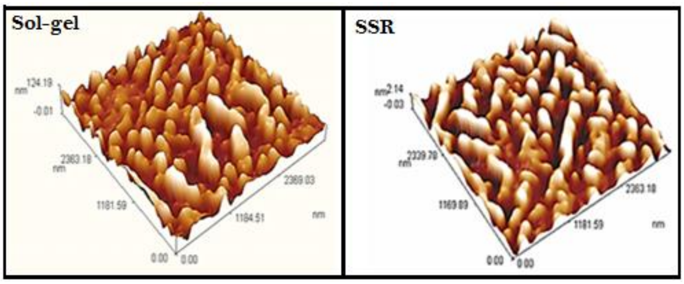

Figure 3AFM image and Granularity accumulation distribution chart of $TlSr_2Ca_2Cu_3O_{9-\delta}$ samples prepare by sol-gel and solid state reaction methods (SSR)

Table 1. The lattice parameters values of $TlSr_2Ca_2Cu_3O_{9-\delta}$.

Method	$T_{c(OFF)}$(K)	$T_{c(ON)}$ (K)	*δ(o₂)*	*O₂*	*a*(A⁰)	*c*(A⁰)	*c/a*	*Avg. Diameter(nm)*
sol-gel	114	121	*0.132*	6.868	3.29	14.879	4.522	92.36
Solid state reaction	111	118	*o.141*	6.859	3.31	14.958	4.519	108.24

CONCLUSION

The high-temperature superconductor (HTSC) $TlSr_2Ca_2Cu_3O_{9-\delta}$ceramic material has been prepared by sol-gel and solid state method. All samples were characterized with X-ray Diffraction Analysis (XRD), Four Point Probe Technique and Atomic Force Microscope (AFM). The results obtained showed that SSR method requires longer time (96 hours) for the heat treatment and several rounds of grinding practices during the preparation process as compared to sol-gel method which is requires (24 hours) only. However, both methods exhibited HTSC ceramics with very good metallic behavior and $T_{C(R=0)}$ above 100K. Both samples consisted of essentially single 1223 phase of tetragonal structures. Sol-gel samples exhibited a bigger crystalline size less cavities and voids as compared to SSR samples. Sol-gel method offers better quality superconducting ceramics with shorter preparation time required. This make it essential for practical use for current leads and current fault limiter. AFM image showed that Average diameter increasing from 92.36nm for sol-gel to 108.24nm solid state method with randomly distributed in all highly compressed samples.

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